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IS 326-20 (1993): Methods of sampling and test for natural and synthetic perfumery materials, Part 20: Determination of boiling (Distillation) range [PCD 18: Natural and Synthetic Fragrance Materials]



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(Reaffirmed 2002)

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भाग 20 क्वथनांक (आसवन) रेंज ज्ञात करना
(दूसरा पुनरीक्षण)

Indian Standard

NATURAL AND SYNTHETIC PERFUMERY
MATERIALS — METHODS OF SAMPLING
AND TEST

PART 20 DETERMINATION OF BOILING (DISTILLATION) RANGE

(*Second Revision*)

UDC 665-57 : 536-423-1

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1952 entitled 'Methods of test for essential oils' and subsequently revised in 1968 as 'Methods of sampling and test for natural and synthetic perfumery materials'. Taking cognizance of the need to incorporate instrumental methods of analysis now in vogue and to facilitate inclusion of additional test methods or changes in the existing test method and also to align these test methods with those specified in corresponding ISO Standards, the Committee decided to revise and split the standard and publish individual test methods as separate standards.

Method of test for boiling (distillation) range as given under **16.7 of IS 326 : 1968** 'Methods of sampling and tests for natural and synthetic perfumery materials (*first revision*)' has been covered in this standard.

This standard is essentially based on ANSI/ASTM D 850-70 'Method for distillation of industrial aromatic hydrocarbons and related materials.'

In reporting the result of a test or analysis, made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

NATURAL AND SYNTHETIC PERFUMERY MATERIALS — METHODS OF SAMPLING AND TEST

PART 20 DETERMINATION OF BOILING (DISTILLATION) RANGE

(*Second Revision*)

1 SCOPE

This standard (Part 20) specifies a method of test for determining the boiling (distillation) range of perfumery materials.

2 DETERMINATION OF DISTILLATION (BOILING) RANGE

2.1 Outline of the Method

The range of temperature between which a liquid boils or the percentage of the material that distil between two specified temperatures is determined. The lower of the two temperatures is the corrected thermometer reading when the first five drops of distillate have been collected, and the upper temperature is the corrected reading when the percentage specified has been collected. This method covers the distillation of aromatic perfumery materials of relatively narrow boiling ranges between 30°C and 250°C.

2.2 Apparatus

2.2.1 Distillation Flask

A standard 200-ml, side-tube, heat-resistant glass distillation flask as shown in Fig. 1, conforming to the following dimensions:

Diameter of bulb, outside, mm	73 ± 1
Diameter of neck, inside, mm	21 ± 1
Height of flask, outside, mm	179 ± 3
Vertical distance from bottom of bulb outside to bottom of vapour-tube opening in neck, mm	120 ± 3
Length of side tube, mm	100 ± 3
Diameter of side tube, outside, mm	7 ± 0.5
Angle of side tube with vertical axis of bulb and neck, deg	75 ± 3

2.2.2 Straight Glass Condenser

The condenser tube may consist of a straight glass tube 600 to 610 mm in length and 12 mm

in inside diameter of standard wall thickness (about 1.25 mm) with the exit end cut off square and ground flat. It shall be set in a cooling trough so that at least 380 mm (15 inch) of the tube is in contact with the water. Clearance between the condenser tube and any parallel side of the trough shall be not less than 19 mm (3/4 inch). The water in the cooling trough shall be maintained at 10 to 20°C. This may be done by adding ice to the water or by circulating chilled water through the trough. The trough shall be so mounted that the condenser tube is set at an angle of 75 deg with the vertical.

2.2.3 Receiver

A graduate of the cylindrical type, of uniform diameter, with a pressed or moulded base and a lipped top. The cylinder shall be graduated to contain 100 ml, and the graduated portion shall be not less than 178 nor more than 203 mm in length. It shall be graduated in single millilitres and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 ml. The overall height of the graduate shall be not less than 208 nor more than 260 mm. The graduations shall not be in error by more than 1 ml at any point on the scale. The bottom 1 ml graduation may be omitted.

2.2.4 Asbestos Board

A sheet of 3 to 6 mm (1/8 to 1/4 inch) hard asbestos board 152 mm (6 inch) square with a circular hole in the centre supported on a circular metal shield enclosing the Bunsen burner, and approximately 50 mm (2 inch) higher than the top of the burner. For materials having low boiling point, the hole shall be 1 inch in diameter; for tests of materials boiling above 100°C but mostly below 145°C the hole shall be 38 mm (1½ inch) in diameter; and for higher boiling materials it shall be 50 mm (2 inch) in diameter.

2.2.5 Heater

An electric heater or a bunsen burner, fully adjustable and capable of giving sufficient heat to distill the product at the required rate. When a bunsen burner is used, the burner shall be adjusted so as to produce an entirely blue flame. In case of dispute concerning results obtained with gas heat versus electric heat, gas heat shall be accepted as the standard.

2.2.6 ¹⁾Thermometer

Thermometer conforming to the following requirements is recommended. When placed in position, the top of the bulb of the thermometer is levelled with the centre of the opening of the outlet tube:

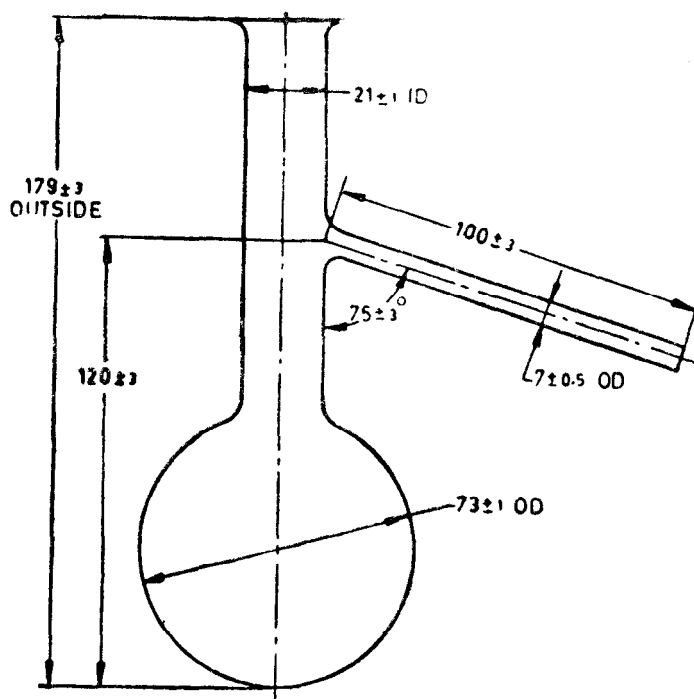
Range	$-2^{\circ}\text{C} + 300^{\circ}\text{C}$
Graduation	1°C
Immersion	Total
Overall length	380 ± 10 mm
Stem diameter	5.5 to 8.0 mm
Bulb shape	Cylindrical
Bulb length	8 to 16 mm
Bulb diameter	Not less than 5.5 and not greater than stem
Length of graduated portion	223 to 254 mm
Distance from bottom of bulb to 0°C	100 to 110 mm

Longer lines at each	5°C
Figured at each	10°C
Expansion chamber	Required
Top finish	Ring
Scale error not to exceed	$\pm 0.5^{\circ}\text{C}$ up to 150°C $\pm 1^{\circ}\text{C}$ above 150°C

Any other thermometer of similar range and accuracy may be used.

2.3 Procedure

2.3.1 Assemble the apparatus as shown in Fig. 2. Mount the flask on the asbestos board of appropriate dimensions, with the side tube extending through a tightly fitting cork stopper about 50 mm (2 inch) into the condenser tube. Support the distillation thermometer in the neck of the flask by means of a cork stopper in such a position that the top of the bulb (or top of contraction bulb if present) is level with the lowest point of juncture between the side tube and the neck and the thermometer is vertical and centrally located in the neck of the flask. When corrections are to be made for emergent stem, suspend an auxiliary thermometer adjacent to the distillation thermometer and with its bulb just below the middle of the emergent mercury thread. Place the burner directly under the centre of the hole in the asbestos board.



All dimensions in millimetres.

FIG. 1 DISTILLATION FLASK

¹⁾ Thermometers with the Institute of Petroleum Designation IP 5 C conform to these requirements.

2.3.2 Carefully measure a 100-ml sample of the material to be tested in the 100-ml graduated cylinder at room temperature and transfer to the distillation flask, draining the cylinder at least 15 s. This is preferably done before mounting the flask in position in order to prevent liquid from entering the side arm. Connect the flask to the condenser and apparatus, assembled as shown in Fig. 2. Do not rinse out the graduated cylinder used to measure the sample for distillation, but place under the lower end of the condenser tube to receive the distillate. Heat the flask slowly, especially after ebullition has begun, so as to allow the mercury column of the thermometer to become fully expanded before the first drop distills over. Regulate the rate of heating so that the ring of condensing vapor on the wall of the flask reaches the lower edge of the side arm in not less than 90 s, and preferably approximately 120 s, from the start of the rise of the vapour ring. The total time from the start of heating until the first drop falls into the receiver should

be not less than 5 nor more than 10 minutes. Avoid major changes in heating rate. Even operation is best gained through experience with the method. When distillation starts, adjust the receiver to allow condensate to flow down its inner wall to prevent loss by spattering; then adjust the heater to continue the distillation at the rate of 5 to 7 ml/min (about 2 drops/s). Maintain this rate, and continue the distillation to dryness.

2.3.3 Record the temperature reading when the first drop of distillate falls into the receiving cylinder. Take additional readings when 5 percent, 10 percent, each additional 10 percent up to 90 percent, 90 percent, and 95 percent of the sample have distilled over. Take a final reading when the liquid just disappears from the bottom of the flask. When testing crude materials, a decomposition point, rather than a dry point, may be obtained. When a decomposition point is reached at the end of a distillation, the temperature will frequently

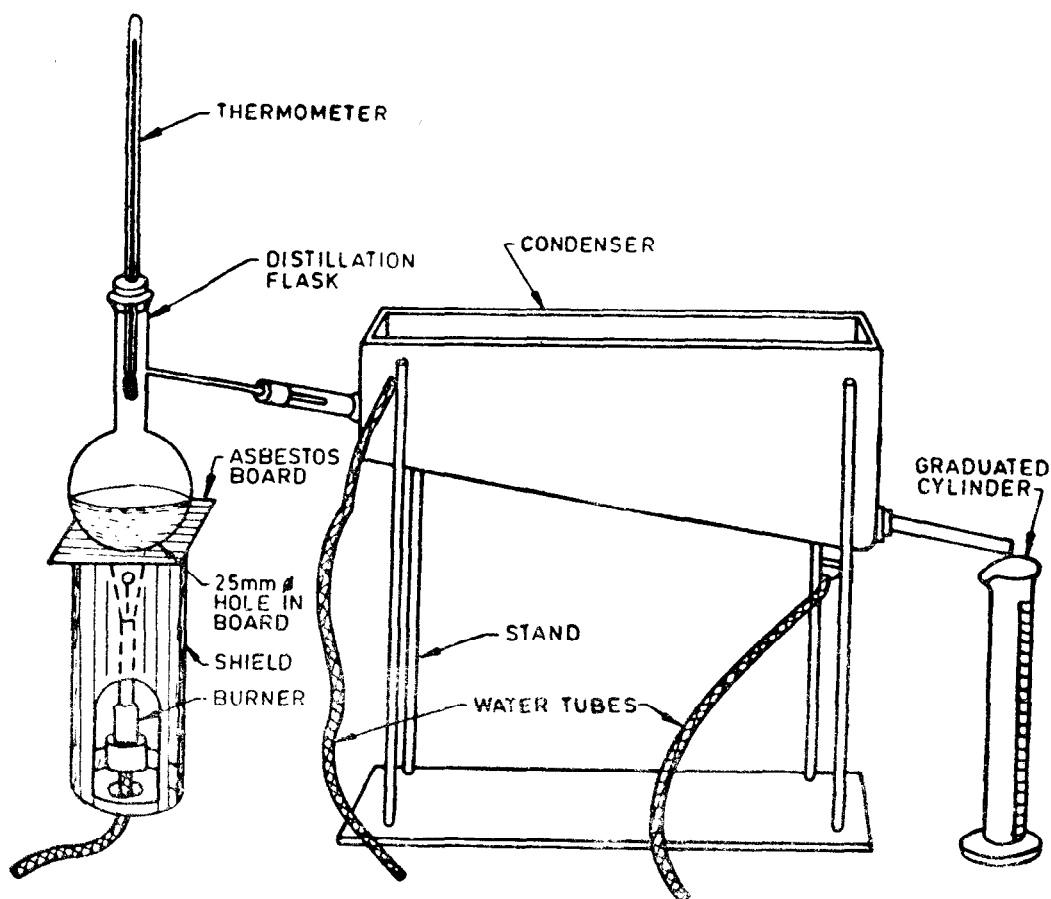


FIG. 2 ASSEMBLY OF APPARATUS FOR DISTILLATION TEST

cease to rise and begin to fall. In these case, take the temperature at the decomposition point as the maximum temperature observed. The decomposition point may also be indicated by the appearance of heavy fumes in the flask. Should that occur, record the temperature at the time the bulb of the flask becomes substantially full of fumes.

2.3.4 Observe and record the following additional data at the time and place of the distillation test.

- a) Number of degree graduations on the emergent stem from top of cork to the graduation at the observed temperature, and the average temperature of the exposed stem.
- b) Correction for inaccuracy of the thermometer.

2.4 Temperature Correction

2.4.1 Corrections of temperature should be applied in the following cases.

2.4.2 When required by the specifications.

2.4.3 When there is any question of compliance with the specification.

2.4.4 When tests of the sample are to be checked against results obtained by another investigator.

2.4.5 Emergent Stem Corrections

Any convenient mercury, etched glass stem thermometer with a range including 20 to 40°C may be used for determining the temperature of exposed stem of the distillation thermometer. Calculate the correction for emergent stem of the thermometer as follows:

$$C = K N (T - t)$$

where

C = number of degrees celsius to be added to the observed thermometer reading;

K = Correction factor for the thermometer = 0.000 154 = difference between coefficients of expansion for glass and for mercury;

N = Number of degree graduations on the stem exposed;

T = Observed reading of the thermometer in degrees celsius; and

t = Average temperature of the emergent stem in degrees celsius.

2.5 Inaccuracy of Thermometer

This correction shall be obtained by calibration of the thermometer used in the test and applied to the observed thermometer reading corrected for emergent stem.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002

Telephone

{ 331 01 31
{ 331 13 75

Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola
CALCUTTA 700054

{ 37 84 99, 37 85 61
{ 37 86 26, 37 86 62

Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036

{ 53 38 43, 53 16 40
{ 53 23 84

Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113

{ 235 02 16, 235 04 42
{ 235 15 19, 235 23 15

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
BOMBAY 400093

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